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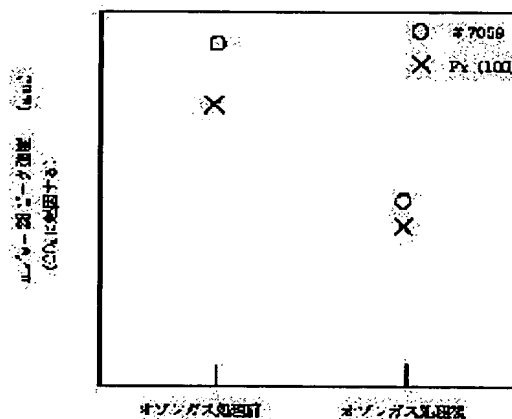
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(54) METHOD OF PROCESSING SUBSTRATE AND PROCESSING DEVICE

(57)Abstract:

PROBLEM TO BE SOLVED: To provide the method of processing a substrate, in which organic compound removed from the substrate does not re-attach it, water molecules are effectively removed from it without increasing processing steps, the steps of drying process are not necessary before forming a thin film on the processed substrate, the deterioration of the roughness or the flatness of the substrate by the processing is little and the processing is not dependent on the material of the substrate and to provide the processing device.

SOLUTION: In the processing method of the substrate, the substrate arranged in a gas tight chamber provided with exhaust means is exposed to ozone gas or/and heated gas. The density of the impurity included in the ozone gas or the heated gas is desirable to be 10ppb or less. Further, the temperature of the heated gas is 80° C or more is desirable.



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CLAIMS

[Claim(s)]

[Claim 1] The art of the base with which the base arranged in the sealed cabin equipped with the exhaust air means at least is characterized by being put to ozone gas.

[Claim 2] The art of the base characterized by putting the base arranged in the sealed cabin equipped with the exhaust air means at least to the heated gas.

[Claim 3] The art of the base with which the base arranged in the sealed cabin equipped with the exhaust air means at least is characterized by being put to ozone gas and the heated gas.

[Claim 4] The art of the base according to claim 1 or 3 with which high impurity concentration contained in said ozone gas is characterized by being 10 or less ppb.

[Claim 5] The art of the base according to claim 2 or 3 with which high impurity concentration contained in said heated gas is characterized by being 10 or less ppb.

[Claim 6] The art of a base given in claims 2 and 3 to which temperature of said heated gas is characterized by being 80 degrees C or more, or any 1 term of 5.

[Claim 7] The art of a base given in claims 2, 3, and 5 characterized by said heated gas being one gas chosen from rare gas, nitrogen gas, ozone gas, or hydrogen gas, or any 1 term of 6.

[Claim 8] The processor of the base characterized by using the art of the base of a publication for claim 1 thru/or any 1 term of 7.

[Claim 9] The processor of the base characterized by forming a thin film at said processing room in the processor of the base which has the sealed cabin and processing room which were equipped with the exhaust air means at least on the base which carried out the art of the base of a publication to claim 1 thru/or any 1 term of 7 in said sealed cabin.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the art and processor of a base. It is related more with the art and processor of a base from which the organic system impurity or/and water molecule which adhered to the detail on the front face of a base are removed.

[0002]

[Description of the Prior Art] In recent years, in the display field and the semi-conductor field, further large capacity and densification are desired. It is necessary to realize a detailed display technique, a detailed circuit technique, etc. in these fields at this time.

[0003] In order to attain the above-mentioned technique, in the reduced pressure (vacuum) ambient atmosphere, the washing process and the etching process are performed for the erector of various kinds [processes / various kinds of / membrane formation processes, etching processes, and cleaning processes] in the atmospheric-air ambient atmosphere to the base.

[0004] Also in which process, to perform a certain processing to a base, it is necessary to hold the base front face before the processing to clarification. That is, it is important to remove the organic system impurity which exists on a base front face, and a water molecule as much as possible. This cleanliness will fall, or as for many properties of a base of having finished processing of each process, variation will become large when unstable. Consequently, there was a problem that the yield of the various products manufactured through each process fell.

[0005] Conventionally, the following technique is known as an approach of coping with the above-mentioned problem.

** How to perform various desiccation processings before forming a thin film on a base after carrying out wet washing of the base using pure water, various alcohol, or various solvents into atmospheric air.

** How to perform various desiccation processings before forming a thin film on a base after carrying out wet etching of the base using various etching solutions into atmospheric air.

** How to carry out dry etching of the base using various plasma into a vacuum.

** How to make generate ozone and put a base to this ozone by decomposing the oxygen contained in atmospheric air, according to the light source of the ultraviolet rays which consist of a spectrum (there is wavelength of 200nm or more besides 184nm) which are not singles, such as a low pressure mercury lamp, into atmospheric air.

** How to deposit one or more layers of thin films on a base, and form a new clarification side on a base into a vacuum.

[0006] However, there are the following problems in the above-mentioned conventional technique.

(1) Before the technique of the above-mentioned ** and ** forms a thin film on a base, its process of various desiccation processings is indispensable, and a manufacturing cost becomes high. Moreover, since it is exposed to atmospheric air after processing, the organic substance and a water molecule will carry out the reattachment to a base front face.

(2) Since the granularity on the front face of a base increases by etching and the surface smoothness of a base also falls, turbulence generates the technique of the above-mentioned ** and ** in the thin film produced on the base, or the structure.

(3) Polymer rye ZESHON of the organic substance removed from the base will occur, and the reattachment of the technique of the above-mentioned ** will be carried out to a base. Moreover, the generating effectiveness of ozone is low under reduced pressure.

(4) Since the process under a vacuum increases, and a manufacturing cost becomes high and the technique of the above-mentioned ** has the quality-of-the-material dependency of a base, its constraint is large.

[0007]

[Problem(s) to be Solved by the Invention] This invention sets it as the 1st purpose to offer the art and processor of a base in which the organic substance removed from the base does not carry out the reattachment.

[0008] Moreover, this invention sets it as the 2nd purpose to offer the art and processor of a base from which a water molecule is efficiently removable, without increasing down stream processing.

[0009] Furthermore, it sets it as the 3rd purpose for the process of various desiccation processings to be unnecessary and to offer the art and processor of a base with few quality-of-the-material dependencies of a base by processing that there are few granularity increases on the front face of a base and falls of surface smoothness, before this invention forms a thin film on the processed base.

[0010]

[Means for Solving the Problem] A summary exists in the art of the base characterized by putting the base arranged in the sealed cabin where this invention was equipped with the exhaust air means at least to ozone gas or/and the heated gas.

[0011]

[Embodiment of the Invention] Below, an operation of each claim concerning this invention is explained.

[0012] In invention concerning claim 1, since the base arranged in the sealed cabin equipped with the exhaust air means at least is put to ozone gas, the amount of survival of the organic system impurity which exists on a base can be reduced.

[0013] In invention concerning claim 2, since it is put to the gas with which the base arranged in the sealed cabin equipped with the exhaust air means at least was heated, the amount of survival of the water molecule which exists on a base can be reduced.

[0014] In invention concerning claim 3, since the base arranged in the sealed cabin

equipped with the exhaust air means at least is put to ozone gas and the heated gas, the amount of survival of an organic system impurity and the amount of survival of a water molecule which exist on a base can be reduced to coincidence. [0015] In invention concerning claim 4, since the high impurity concentration contained in said ozone gas is 10 or less ppb, the amount of survival of the organic system impurity which exists on a base can be lessened further.

[0016] In invention concerning claim 5, since the high impurity concentration contained in said heated gas is 10 or less ppb, the amount of survival of the water molecule which exists on a base can be lessened further.

[0017] In invention concerning claim 6, since the temperature of said heated gas is 80 degrees C or more, the effectiveness of reducing the amount of survival of the water molecule which exists on a base increases remarkably. Consequently, shortening of the processing time of a base is also attained.
 [0018] In invention concerning claim 7, since said heated gas is one gas chosen from rare gas, nitrogen gas, ozone gas, or hydrogen gas, the art of a base with a high removal operation of the water molecule which exists on a base is obtained.

[0019] In invention concerning claim 8, since the art of the base of a publication was used for claim 1 thru/or any 1 term of 7, the processor of the base which can reduce the amount of survival of the organic system impurity which exists on a base, or/and the amount of survival of a water molecule is obtained.

[0020] In invention concerning claim 9, in the processor of the base which has the sealed cabin and processing room which were equipped with the exhaust air means at least, in order to form a thin film at said processing room on the base which carried out the art of the base of a publication to claim 1 thru/or any 1 term of 7 in said sealed cabin, the thin film by which structure was stabilized is obtained.

[0021] Hereafter, the example of an embodiment of this invention is explained with reference to a drawing.

[0022] (Sealed cabin equipped with the exhaust air means at least) The sealed cabin concerning this invention has the exhaust air means which can eliminate the ozone gas introduced into the airtight interior of a room, and the heated gas, as shown in drawing 2 , drawing 3 , or drawing 10 . Various general-purpose pumps may be used as an exhaust air means. When making the airtight interior of a room into the bottom of reduced pressure, various vacuum pumps are used suitably. Especially when there are many quantities of gas flow to introduce, multistage connection is made and two or more pumps are used. For example, the combination of a molecular drag pump (Daikin make, DMS600ACR) + mechanical booster-pump (made in [Edwards], QMB500) + dry pump (made in Edwards, QDP80) is mentioned. In this combination, continuous duty becomes possible by quantity-of-gas-flow 50SLM.

[0023] As an ingredient which constitutes the wall of a sealed cabin, SUSs (316L, 304L, etc.), aluminum, and nickel alloys (Hastelloy etc.) are mentioned, for example. Especially to introduce ozone gas into a sealed cabin, the wall of a sealed cabin needs to consist of high ingredients of ozone proof gas nature. As such an ingredient, 203-/SUS-316of Fe(s) L (SUS-316L in which 2OFe3 dozens of nm film was formed on the front face) is suitable, for example. It is advantageous from the point that the quality of the material does not reform by O3, and O3 does not

deactivate compared with aluminum.

[0024] Moreover, when introducing the gas with which it was heated other than O₃, as an ingredient which constitutes the wall of a sealed cabin, aluminum with which /SUS-316L (what prepared 2OCr₃ film and 2OFe₃ film in electrolytic polishing or the front face of SUS-316L which carried out compound electrolytic polishing), and surface roughness Ra were ground by about 0.1 micrometers, for example (Cr 2O₃, Fe₂O₃) is used suitably. The point with few impurity gas evolutions from the ingredient itself which constitutes a front face is excellent.

[0025] It is more desirable to have made it the gas which the exhaust air means countered mostly with the location connected to a sealed cabin, and prepared the location where a gas feed system is connected to a sealed cabin, and was introduced into the airtight interior of a room serve as a turbulent flow. It excels from the point that the effectiveness of this invention is not spoiled, without gas piling up, when this arrangement is adopted. For example, arrangement as shown in drawing 2 or drawing 3 is mentioned.

[0026] The TDS (Thermal Desorption Spectroscopy, temperature-programmed-desorption gas-analysis method) chemical laboratory was established in the sealed cabin mentioned above through the gate valve. In the TDS chemical laboratory, the organic system impurity and water molecule adhering to a base front face were made to break away by applying heat to a substrate, and the analysis apparatus which mentions the balking gas later estimated quantitatively.

[0027] The TDS chemical laboratory is equipped with the exhaust air means, and the exhaust air means is suitably chosen like a sealed cabin by the ambient pressure force which carries out assay of the organic system impurity adhering to a base front face, or the water molecule.

[0028] When the pressure of the TDS analysis interior of a room measured near the atmospheric pressure, Hitachi Tokyo Electronics TDS equipment (UG-21) was attached in the chemical laboratory (drawing 2). Evaluation of the organic system impurity and water molecule which were desorbed from the base front face was carried out using APIMS (Atmospheric Pressure Ionization Mass Spectrometer, atmospheric-pressure-ionization-mass-spectrometry equipment) attached to TDS equipment. Moreover, Ar and N₂ grade was suitably introduced as carrier gas during analysis.

[0029] On the other hand, when measuring under reduced pressure, the TDS equipment made from electronic science (EMD-WA1000K) was attached in the chemical laboratory (drawing 3). Evaluation of the organic system impurity and water molecule which were desorbed from the base front face was carried out using QMS (Quadrupole Mass Analyzer, quadrupole mass spectrograph) attached to TDS equipment.

[0030] (Base) As a base, glass substrates (HOUKEI acid alkali free glass, soda glass, quartz, etc.), Si wafer substrates (FZ (100), CZ (100), etc.), and ceramic substrates (aluminum₂ O₃, SiC, etc.) are mentioned, for example. Moreover, what prepared various coats on these substrates is included. As various coats, aluminum, Mo, Ta, SiN, TiN, a-Si, etc. are mentioned, for example. Furthermore, it is an example of the base which carries out the laminating of these film and what arranged an electronic device or a circuit, the magnetic cell or the circuit and the

optical element, or the circuit on said substrate requires for this invention by carrying out configuration processing.

[0031] As a base which has an electronic device or a circuit, they are TFT (Thin Film Transistor), MOSFET (Metal Oxide Semiconductor Field Emission Transistor), or Diode Capacitor, for example. What was prepared is mentioned. When producing, the LCD cel (Liquid Crystal Display Cell) LCD, i.e., the passive-matrix mold, which has the segment electrode and common electrode which were formed especially with the transparent electrode, the LCD cel (Liquid Crystal Display Cell) LCD, i.e., the active-matrix mold, which has a TFT (ThinFilm Transistor) circuit, the art and processor of the base concerning this invention are used suitably.

[0032] As a base which has a magnetic cell or a circuit, a magnetic disk, the magnetic head, and a magnetic card are mentioned, for example. As a base which has an optical element or a circuit, various kinds of carrier luminescence devices and the optical (optical MAG) disk which are represented by LED and semiconductor laser, for example are mentioned.

[0033] (Ozone gas) The ozone gas concerning this invention is the mixed gas which consisted of O₃, O₂, and N₂, and what has O₃ concentration in the range of 1 ppm – 1000 ppm (O₃:(O₂+N₂) =1:10⁶–10³) is used suitably. Gas constituents other than O₃, O₂, and N₂ are the impurities contained in ozone gas, for example, H₂O, and CO₂ and CH₄ are mentioned. When referred to as 1000 or less ppb, since the amount of detection of the organic system gas impurity at the time of TDS measurement decreases greatly and the removal effectiveness of an organic system gas impurity becomes remarkable, the high impurity concentration contained in this ozone gas is desirable. Moreover, since it is saturated with a condition with few amounts of detection of an organic system gas impurity and can judge that the amount of organic system gas impurities which exists in a base front face became the minimum value mostly when this high impurity concentration is set to 10 or less ppb, it is more desirable.

[0034] To what improved a part of O₃ generation machine (the Sumitomo Precision Products make, SG-01A1) of marketing arranged near the sealed cabin, the ozone gas in this invention supplied O₂ and N₂ of 1 or less ppb, and manufactured high impurity concentration. The high impurity concentration of the obtained ozone gas is 1 or less ppb, and was introduced into the sealed cabin through piping of an ozone proof gas specification. Moreover, the high impurity concentration contained in the ozone gas which content high impurity concentration adds in ozone gas of 1 or less ppb, and introduces a suitable quantity of an impurity in a sealed cabin was changed in the range of 100 ppm – 1ppb from the impurity addition system which becomes the piping section which connects a sealed cabin to O₃ generation machine from the gas dilution system which combined with multistage N₂ gas supply system which added 10 ppm of known impurities, and the massflow controller.

[0035] (Heated gas) The type of gas which is easy to tell heat to a base as a heated gas concerning this invention through a gas, and can remove a water molecule from a base front face is desirable. From this reason, one gas chosen from rare gas (helium, Ar, etc.), nitrogen gas, ozone gas, or hydrogen gas is used suitably.

[0036] It is the impurity with which gas constituents other than the heated each gas are contained in the each heated gas, for example, H₂O, and CO₂ and CH₄ are mentioned. When the heated gas is nitrogen gas, the high impurity concentration contained in nitrogen gas Content high impurity concentration in the piping section which connects the supply system and sealed cabin of nitrogen gas of 1 or less ppb From the impurity addition system which consists of a gas dilution system which combined with multistage the another supply system and another massflow controller of the nitrogen gas which added 100 ppm of known impurities, content high impurity concentration adds a suitable quantity of an impurity to the nitrogen gas of 1 or less ppb. The high impurity concentration contained in the nitrogen gas introduced into a sealed cabin was changed in the range of 100 ppm – 1ppb. When referred to as 1000 or less ppb, since the amount of detection of H₂O at the time of TDS measurement decreases greatly and the removal effectiveness of H₂O becomes remarkable, the high impurity concentration contained in this heated each gas is desirable. Moreover, since it is saturated with a condition with few amounts of detection of H₂O and can judge that the amount of H₂O which exists in a base front face became the minimum value mostly when this high impurity concentration is set to 10 or less ppb, it is more desirable.

[0037] As an approach of applying heat to a gas, a heater is attached in gas installation piping to a sealed cabin, for example, and the approach of heating indirectly through piping is mentioned. When it considers as 50 degrees C or more, since the amount of detection of H₂O at the time of TDS measurement decreases greatly and the removal effectiveness of H₂O becomes remarkable, the temperature of the heated each gas is desirable. Moreover, since it is saturated with a condition with few amounts of detection of H₂O and can judge again that the amount of H₂O which exists in a base front face became the minimum value mostly when this temperature is made into 80 degrees C or more, it is more desirable.

[0038] (Processor of the base which has the sealed cabin and processing room which were equipped with the exhaust air means at least) The processing room is established in the sealed cabin which mentioned above the processor of the base concerning this invention through the gate valve. The processing room is equipped with the exhaust air means at least. The exhaust air means is suitably chosen by airtight indoor working pressure like a sealed cabin. The exhaust air means suitably depended for and chosen as airtight indoor working pressure independently depending on the case may be established. Therefore, there is especially no limit in the internal pressure of a processing room.

[0039] Although a thin film is formed in the processing interior of a room on the base which finished predetermined processing in the sealed cabin, the thin film formation approaches may be any. For example, as for the case under atmospheric pressure, in under reduced pressure, such as the applying method and the galvanizing method, vacuum deposition, a spatter, a CVD method, etc. are mentioned.

[0040] Although there is no limit in the ingredient of the thin film formed, in case a thin film is formed, a base may be suitably heated or cooled from the purpose which controls many properties, adhesion, etc. of a thin film. Moreover, electric

field or/and a magnetic field may be introduced in the space in which a thin film is formed.

[0041] (The evaluation approach of a base) Below, the evaluation approach of the base applied to this invention using drawing 2 and drawing 3 R> 3 is explained.

[0042] (1) When the pressure of the TDS analysis interior of a room was near the atmospheric pressure, the ** bases I and II were set to the pivotable base electrode holder of a sealed cabin.

** Base I was moved to the TDS equipment which impurity gas concentration other than Ar is purged by the high grade Ar of 1 or less ppb, and always has it in a pure ambient atmosphere.

** Carrying out 2 l/min supply of the Ar, Base I was heated from a room temperature to 500 degrees C, and the carrier Ar gas by which the gas from which it was desorbed in the process is contained was analyzed by APIMS.

** Processing according Base I to ozone gas or/and the heated gas was performed to Bases I and II after moving to a sealed cabin.

** Like the above-mentioned ** and **, Base II was moved to TDS equipment and it analyzed by APIMS.

[0043] (2) When the pressure of the TDS analysis interior of a room was under reduced pressure, the **' bases I and II were set to the pivotable base electrode holder of a sealed cabin.

** Base I was moved to the TDS equipment beforehand decompressed to 2×10^{-9} Torr after decompressing 'sealed cabin to 1×10^{-6} Torr.

** The 'base I was heated from a room temperature to 500 degrees C, and the gas from which it was desorbed in the process was analyzed by QMS.

** Processing according the 'base I to ozone gas or/and the heated gas was performed to Bases I and II after moving to a sealed cabin.

** Like 'above-mentioned **' and **, Base II was moved to TDS equipment and it analyzed by APIMS.

[0044] (The evaluation approach of a thin film) As evaluation criteria of aluminum thin film concerning this invention, specific resistance, a hillock, hot-pure-water oxidation, and crystallinity are mentioned. Each of these evaluation approaches are indicated below.

[0045] [Specific resistance] specific resistance (ohm-cm) was computed from the product of sheet resistance (ohm/**) and thickness (nm). Sheet resistance was measured to the thin film immediately after membrane formation (on a base) using HA-6100/RG-1000E made from NAPUSON. Then, patterning processing of the thin film was carried out, and the thickness of a sheet resistance measurement part was measured by DEKTAK-3030 made from ULVAC.

[0046] The [hillock] hillock was measured with the following procedures.

** The thin film immediately after membrane formation (on a base) was observed with the optical microscope, and the photograph was taken.

** Annealing treatment of the thin film (every base) of the above-mentioned ** was carried out at 400 degrees C under N2 ambient atmosphere for 2 hours.

** The thin film (on a base) which finished processing of the above-mentioned ** was observed with the optical microscope, and the photograph was taken.

** The number of the hillocks in the photograph (scale factor: 825) of the above-

mentioned ** and ** was measured respectively, it asked for the difference, and the existence of the increment in a hillock per unit area was investigated. Hillock size measured the thing about hundreds of nm or more.

[0047] [Hot-pure-water oxidation] hot-pure-water oxidation was measured with the following procedures.

** The thin film after thickness measurement (on a base) was immersed in 80-degree C ***** for 20 minutes by the above-mentioned specific resistance.

** The thickness of a thin film (on a base) which finished processing of the above-mentioned ** was measured. When the thickness after immersion increased from immersion before, aluminum oxidized, it changed to AlOX and it was judged that the volume increased. The existence of this oxidation state was separately checked using XPS (Xray Photoelectron Spectroscopy, X-ray photoelectron spectroscopy).

[0048] [Crystalline] crystallinity measured diffraction reinforcement with the Riken X diffraction measuring device after cutting into 3cm angle the base to which the thin film immediately after membrane formation adhered.

[0049]

[Example] Although an example is raised to below and this invention is explained more to a detail, this invention is not limited to these examples.

[0050] (Example 1) In this example, the base was arranged in the airtight interior of a room shown in drawing 2 , and the amount of survival of the organic system impurity which exists on a base before and after putting to ozone gas [near the atmospheric pressure] was investigated in the TDS chemical laboratory established in the sealed cabin through the gate valve. In the TDS chemical laboratory, it was made to secede from the organic system impurity adhering to a base front face, and applying heat to a substrate estimated the balking gas quantitatively using APIMS. As a base, Si wafer (FZ (100)) or glass (#7059) was used.

[0051] Below, it explains according to an experiment procedure.

(1) Processing shown in ** and ** of a degree was performed one by one, and the base of two sheets was washed.

** Ultrapure water (specific resistance: 18 M omega) immersion of the cassette (quality of the material: tefzel) containing a base was carried out, and it applied megasonic (0.8MHz) one for 10 minutes into ultrapure water.

** Spin desiccation (for 850rpm and 2 minutes) of the base which finished processing of the above-mentioned ** was carried out.

(2) After inserting in the airtight interior of a room the cassette containing the base of two sheets which finished processing of the above (1), the base of one sheet was moved to the TDS chemical laboratory, while it had been unsettled, it made it secede from the organic system impurity adhering to a base front face, and measured the amount of balking by APIMS.

(3) Other bases of one sheet were put to ozone gas by introducing ozone gas into the airtight interior of a room the following condition.

- distributed gas: -- high-impurity-concentration [of O3100 ppm/O2:N2=1:4 and ozone gas]: -- internal pressure [of 1 or less ppb and a sealed cabin]: -- 760Torr and supply time amount: -- moved the base which finished processing of

10min (4) above (3) to the TDS chemical laboratory, it was made to secede from the organic system impurity which adhered to the base front face like the above (2), and the amount of balking was measured.

[0052] Drawing 1 is as a result of [of the amount of organic system impurities (mass number 28) which seceded from the base front face before and after putting to ozone gas] measurement. Drawing 1 showed that it was not dependent on the quality of the material of a base, and the organic system impurity was removed on the front face of the base put to ozone gas.

[0053] (Example 2) In this example, a base is arranged in the airtight interior of a room shown in drawing 3, and the point which put the base to ozone gas in the sealed cabin under reduced pressure differs from an example 1. The TDS chemical laboratory was also made into the bottom of reduced pressure of a sealed cabin and this level, it was made to secede from the organic system impurity adhering to a base front face, and applying heat to a substrate estimated the balking gas quantitatively using QMS. Other points presupposed that it is the same as that of an example 1.

[0054] Below, it explains according to an experiment procedure.

(1) The base of two sheets was washed like the process (1) of an example 1.

(2) After inserting in the airtight interior of a room the cassette containing the base of two sheets which finished processing of the above (1), the airtight interior of a room was decompressed to 1×10^{-6} Torr.

(3) The base of one sheet was moved to the TDS chemical laboratory beforehand decompressed to 2×10^{-9} Torr, while it had been unsettled, it made it secede from the organic system impurity adhering to a base front face, and measured the amount of balking by QMS.

(4) Other bases of one sheet were put to ozone gas by introducing ozone gas into the airtight interior of a room the following condition.

— Move the base which finished processing of 10min (5) above (4) to the TDS chemical laboratory beforehand decompressed to 2×10^{-9} Torr. distributed gas: — high-impurity-concentration [of O3100 ppm/O2:N2=1:4 and ozone gas]: — internal pressure [of 1 or less ppb and a sealed cabin]: — 700Torr and supply time amount: — It was made to secede from the organic system impurity which adhered to the base front face like the above (2), and the amount of balking was measured.

[0055] Drawing 4 is as a result of [of the amount of organic system impurities (mass number 28) which seceded from the base front face before and after putting to ozone gas] measurement. Drawing 4 showed that it was not dependent on the quality of the material of a base, and the organic system impurity was removed on the front face of the base put to ozone gas under reduced pressure.

[0056] (Example 3) In this example, the base was arranged in the airtight interior of a room shown in drawing 2, and the amount of survival of the water molecule which exists on a base before and after putting to the gas heated [near the atmospheric pressure] was investigated in the TDS chemical laboratory established in the sealed cabin through the gate valve. 50-degree C nitrogen gas was used as a heated gas.

[0057] In the TDS chemical laboratory, the water molecule adhering to a base front

face was made to break away, and applying heat to a substrate estimated the balking gas quantitatively using APIMS. As a base, Si wafer (FZ (100)) or glass (#7059) was used. Other points presupposed that it is the same as that of an example 1.

[0058] Below, it explains according to an experiment procedure.

(1) The base of two sheets was washed like the process (1) of an example 1.

(2) After inserting in the airtight interior of a room the cassette containing the base of two sheets which finished processing of the above (1), while it had been unsettled, moved to the TDS chemical laboratory, and the water molecule adhering to a base front face was made to break away, and the base of one sheet measured the amount of balking by APIMS.

(3) Other bases of one sheet were put to nitrogen gas by introducing nitrogen gas into the airtight interior of a room the following condition.

– Distributed gas : N₂ (temperature of 50 degrees C, flow rate 50SLM)

– high-impurity-concentration [of nitrogen gas]: -- internal pressure [of 1 or less ppb and a sealed cabin]: -- 760Torr and supply time amount: -- moved the base which finished processing of 10min (4) above (3) to the TDS chemical laboratory, the water molecule which adhered to the base front face like the above (2) was made to break away, and the amount of balking was measured.

[0059] Drawing 5 is as a result of [of the water molecular weight (mass number 18) which seceded from the base front face before and after putting to the heated nitrogen gas] measurement. Drawing 5 showed that it was not dependent on the quality of the material of a base, and the water molecule was removed on the front face of the base put to the heated nitrogen gas.

[0060] (Example 4) In this example, a base is arranged in the airtight interior of a room shown in drawing 3, and the point put to the nitrogen gas which had the base heated in the sealed cabin under reduced pressure differs from an example 3. The TDS chemical laboratory was also made into the bottom of reduced pressure of a sealed cabin and this level, the water molecule adhering to a base front face was made to break away, and applying heat to a substrate estimated the balking gas quantitatively using QMS. Other points presupposed that it is the same as that of an example 3.

[0061] Below, it explains according to an experiment procedure.

(1) The base of two sheets was washed like the process (1) of an example 1.

(2) After inserting in the airtight interior of a room the cassette containing the base of two sheets which finished processing of the above (1), the airtight interior of a room was decompressed to 1×10^{-4} Torr.

(3) While it had been unsettled, moved to the TDS chemical laboratory beforehand decompressed to 2×10^{-9} Torr, and the water molecule adhering to a base front face was made to break away, and the base of one sheet measured the amount of balking by QMS.

(4) Other bases of one sheet were put to nitrogen gas by introducing nitrogen gas into the airtight interior of a room the following condition.

– Distributed gas : N₂ (temperature of 50 degrees C, flow rate 50SLM)

– high-impurity-concentration [of nitrogen gas]: -- internal pressure [of 1 or less ppb and a sealed cabin]: -- 700Torr and supply time amount: -- moved the

base which finished processing of 10min (5) above (4) to the TDS chemical laboratory beforehand decompressed to 2×10^{-9} Torr, the water molecule which adhered to the base front face like the above (2) was made to break away, and the amount of balking was measured.

[0062] Drawing 6 is as a result of [of the water molecular weight (mass number 18) which seceded from the base front face before and after putting to the heated nitrogen gas] measurement. Drawing 6 showed that it was not dependent on the quality of the material of a base, and the water molecule was removed on the front face of the base put to the nitrogen gas heated under reduced pressure.

[0063] Although nitrogen gas was used as a heated gas in the examples 3 and 4, even if it used rare gas (for example, helium, Ar, Xe, etc.), ozone gas, or hydrogen gas instead of nitrogen gas, it was checked separately that there is the same operation. Especially when ozone gas is adopted as a heated gas, an example 1, 2 and an example 3, or effectiveness of 4 can be realized to coincidence. That is, since the organic system impurity and water molecule which exist on the front face of a base were removable to coincidence, shortening of down stream processing could be attained and the art of the possible base of low-cost-izing was obtained.

[0064] (Example 5) In this example, it differs from an example 1 in that the high impurity concentration contained in ozone gas was changed in the range of 1 ppm – 1ppb. Glass (#7059) was used as a base. Other points presupposed that it is the same as that of an example 1.

[0065] Drawing 7 is the result of measuring the high impurity concentration contained in ozone gas, and the amount of the organic system impurity which remains on the base front face after processing. Since the amount of the organic system impurity which secedes from a base front face at the time of TDS measurement decreased rapidly from drawing 7 when referred to as 1 ppm or less, it turned out that the amount of the organic system impurity which remains on a base front face decreases. Moreover, since the inclination to which the amount of the organic system impurity which secedes from a base front face decreases eased sharply when referred to as 10 or less ppb, it was judged that the amount of the organic system impurity which remains on a base front face approached the minimum value mostly.

[0066] (Example 6) In this example, it differs from an example 3 in that the high impurity concentration contained in the heated nitrogen gas was changed in the range of 1 ppm – 1ppb. Glass (#7059) was used as a base. Other points presupposed that it is the same as that of an example 3.

[0067] Drawing 8 is the result of measuring the amount of the high impurity concentration contained in the heated nitrogen gas, and the water molecule which remains on the base front face after processing. Since the amount of the water molecule which secedes from a base front face at the time of TDS measurement decreased rapidly from drawing 8 when referred to as 1 ppm or less, it turned out that the amount of the owner water molecule which remains on a base front face decreases. Moreover, since the inclination to which the amount of the water molecule which secedes from a base front face decreases eased sharply when referred to as 10 or less ppb, it was judged that the amount of the water molecule which remains on a base front face approached the minimum value mostly.

[0068] (Example 7) In this example, it differs from an example 3 in that whenever [stoving temperature / of nitrogen gas] was changed in room temperature (about 20 degrees C) -400 degree C. Glass (#7059) was used as a base. Other points presupposed that it is the same as that of an example 3.

[0069] Drawing 9 is the result of measuring the temperature of nitrogen gas, and the amount of the water molecule which remains on the base front face after processing. Since the amount of the water molecule which secedes from a base front face decreased rapidly from drawing 9 when it considered as 50 degrees C or more, it turned out that the amount of the water molecule which remains on a base front face decreases. Moreover, since the inclination to which the amount of the water molecule which secedes from a base front face decreases eased sharply when it considered as 80 degrees C or more, it was judged that the amount of the water molecule which remains on a base front face approached the minimum value mostly.

[0070] (Example 8) In this example, the thin film was formed at the processing room on the base put to the gas heated in the sealed cabin shown in drawing 10 in the front face, and many properties of the produced thin film were investigated.

[0071] As a heated gas, glass (#7059) was used as a base and aluminum was used for nitrogen gas as a thin film. The evaluated film properties are specific resistance, a hillock, hot-pure-water oxidation, and crystallinity.

[0072] Below, it explains according to an experiment procedure.

(1) The same base washing as the process (1) of an example 1 was performed.

(2) The cassette containing the base which finished processing of the above (1) was inserted in the sealed cabin.

(3) Evacuation of the airtight interior of a room was carried out to 1×10^{-4} Torr.

(4) As membrane formation pretreatment, by [which adjust exhaust air conductance] having been heated, nitrogen gas (temperature [of 150 degrees C], flow rate 50SLM) installation was carried out, the airtight indoor pressure was set to 5Torr(s), and purge processing was carried out for 1 minute.

(5) After suspending the nitrogen gas supply to a sealed cabin, evacuation of the airtight interior of a room was carried out to 1×10^{-2} Torr.

(6) The above (4) The process of - (5) was repeated 4 times.

(7) Evacuation of the airtight interior of a room was carried out to 5×10^{-7} Torr.

(8) The base was moved to the processing room which has carried out evacuation to 5×10^{-7} Torr separately from the sealed cabin.

(9) At the processing room, high-frequency power (or direct current power of power-flux-density 4 W/cm²) with a frequency [of power-flux-density 1.5 W/cm²] of 13.56MHz was supplied to the cathode, the plasma was generated, and the aluminum film with a thickness of 200nm was formed on the heated base (150 degrees C).

[0073] The above (1) The sample produced according to the process of - (9) was called **** 8.

[0074] (Example 1 of a comparison) In this example, pretreatment (6) of the base in an example 8, i.e., process [of an example 8] (3) -, was deleted. Other points presupposed that it is the same as that of an example 8.

[0075] The sample produced by this example was called the trial ratio 1.

[0076] Table 1 is as a result of [which were performed to each sample produced in the example 6 mentioned above and the example 1 of a comparison] specific resistance, a hillock, hot-pure-water oxidation, and as a result of [about crystallinity / evaluation].

[0077]

[Table 1]

評価項目	試実 6	試比 6
比抵抗	2. 8 $\mu\Omega$	3. 0 $\mu\Omega$
ヒロック	確認されず	確認された
温純水酸化	酸化されず	酸化を確認
結晶性	50 cps/nm	2 cps/nm

[0078] Table 1 showed that it was the thin film by which the direction of **** 6 was stabilized compared with the trial ratio 1. Therefore, in the display field, the semi-conductor field, etc., when various thin films were produced, it was judged by using the art of the base concerning this invention that it was raised by the stability of membrane structure.

[0079] Moreover, generally, since crystallinity had been improved, it was judged that the art of the base concerning this invention almost had neither the granularity increase in a processing side, nor the fall of surface smoothness.

[0080] Although this example examined the art of the base applied to this invention using the easy monolayer of structure, it cannot be overemphasized that it is effective also in the process which produces a semiconductor device, TFT-LCD, etc. of the various kinds which have multilayer structure and complicated structure.

[0081]

[Effect of the Invention] According to this invention, as explained above, a water molecule can be removed efficiently, without the organic substance removed from the base not carrying out the reattachment, but increasing down stream processing, before forming a thin film on the processed base, the process of various desiccation processings is unnecessary, and the art and processor of a base with few quality-of-the-material dependencies of a base are obtained by processing that there are few granularity increases on the front face of a base and falls of surface smoothness.

[Translation done.]

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is the graph which shows the measurement result of the amount of the organic system impurity which seceded from the base front face before and after putting to ozone gas [near the atmospheric pressure concerning the example 1 of this invention].

[Drawing 2] It is the typical sectional view of the processor of the base which consists of a sealed cabin concerning the example 1 of this invention, and a TDS chemical laboratory.

[Drawing 3] It is the typical sectional view of the processor of the base which consists of a sealed cabin concerning the example 2 of this invention, and a TDS chemical laboratory.

[Drawing 4] It is the graph which shows the measurement result of the amount of the organic system impurity which seceded from the base front face before and after putting to ozone gas under reduced pressure concerning the example 2 of this invention.

[Drawing 5] It is the graph which shows the measurement result of the amount of the water molecule which seceded from the base front face before and after putting to the nitrogen gas heated [near the atmospheric pressure concerning the example 3 of this invention].

[Drawing 6] It is the graph which shows the measurement result of the amount of the water molecule which seceded from the base front face before and after putting to the nitrogen gas heated under reduced pressure concerning the example 4 of this invention.

[Drawing 7] It is the graph which shows the measurement result of the high impurity concentration contained in the ozone gas concerning the example 5 of this invention, and the amount of the organic system impurity which remains on the base front face after processing.

[Drawing 8] It is the graph which shows the measurement result of the amount of the high impurity concentration contained in the heated nitrogen gas concerning the example 6 of this invention, and the water molecule which remains on the base front face after processing.

[Drawing 9] It is the graph which shows the measurement result of the temperature of the nitrogen gas concerning the example 7 of this invention, and the amount of the water molecule which remains on the base front face after processing.

[Drawing 10] It is the typical sectional view of the processor of the base which consists of a sealed cabin concerning the example 8 of this invention, and a

processing room.

[Description of Notations]

200,300,900 Sealed cabin,
201, 301, 901 TDS equipment,
202 APIMS,
203, 303, 903 O3 generation machine,
204, 304, 904 ** gas-generator style,
205, 305, 905 Gas diluter,
206, 306, 906 Standard chemical cylinder,
207, 307, 907 Base I
208, 308, 908 Base II,
209, 309, 909 Base electrode holder (cassette),
210, 310, 320, 910, 931 Turbo molecular pump
211, 311, 911 Gate valve,
212 312 Base at the time of TDS measurement,
213 313 Base electrode holder of TDS equipment,
214 Infrared Lamp,
215 High Grade Ar Installation Rhine,
216 Ar Rhine between TDS Equipment and APIMS,
217, 218, 317, 318, 917, 918 High grade N of two lines
219, 319, 919 High grade O2 line,
302 QMS,
314 Infrared Installation Equipment,
912 Base of Processing Interior of a Room,
930 Processing Room,
932 Ar Rhine,
933 Aluminum Target,
934 A substrate electrode holder-cum-a cathode.

[Translation done.]

*** NOTICES ***

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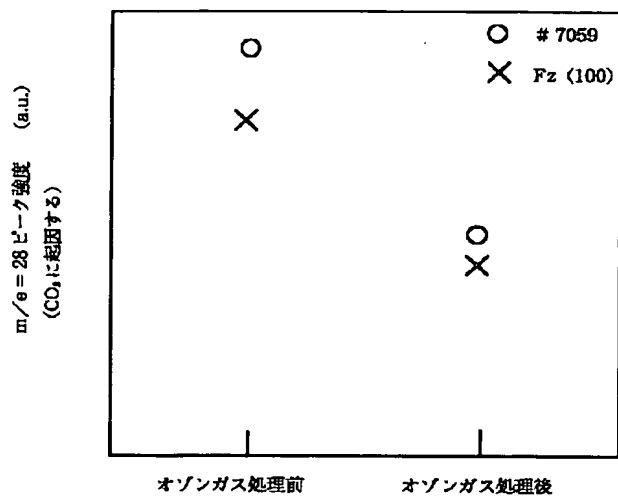
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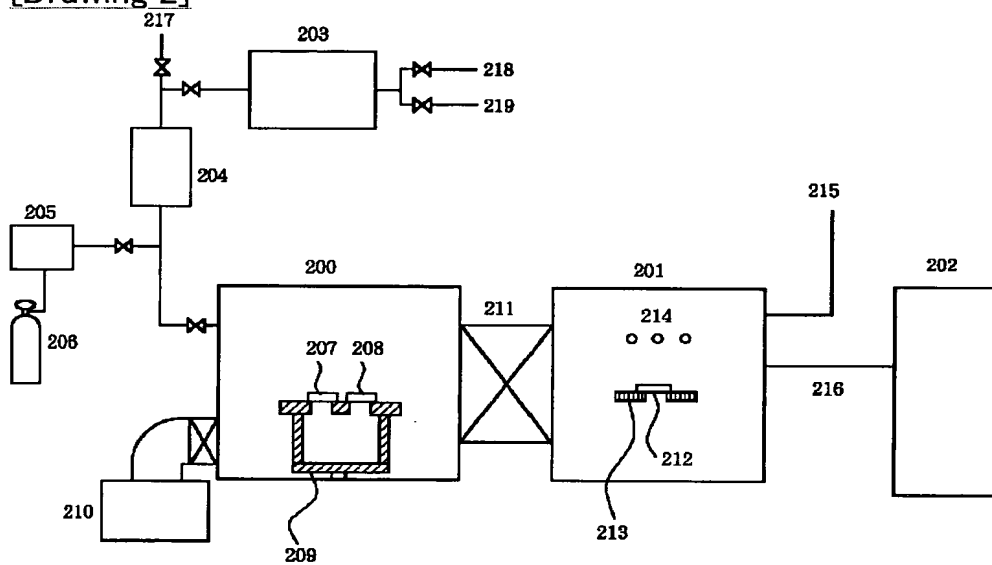
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DRAWINGS

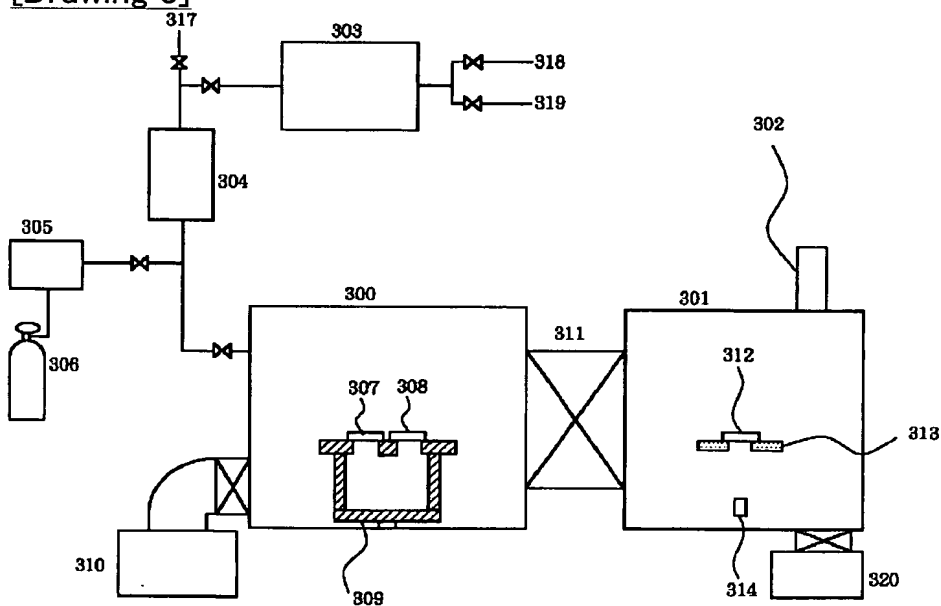
[Drawing 1]



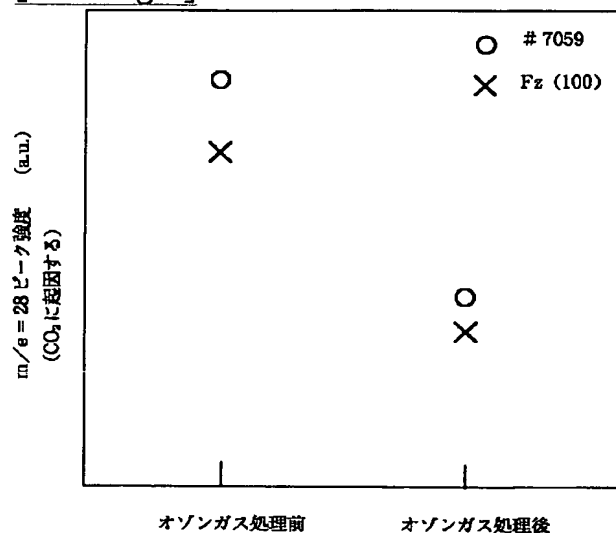
[Drawing 2]



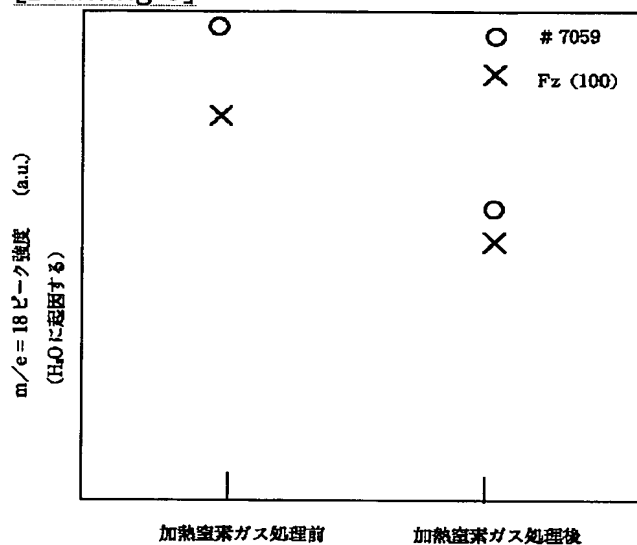
[Drawing 3]



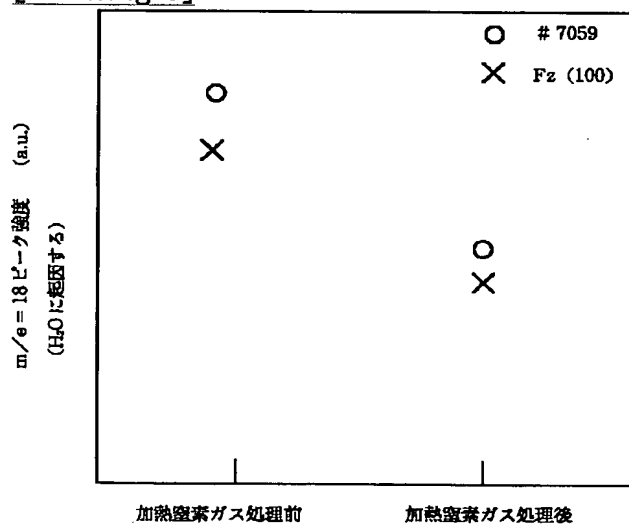
[Drawing 4]



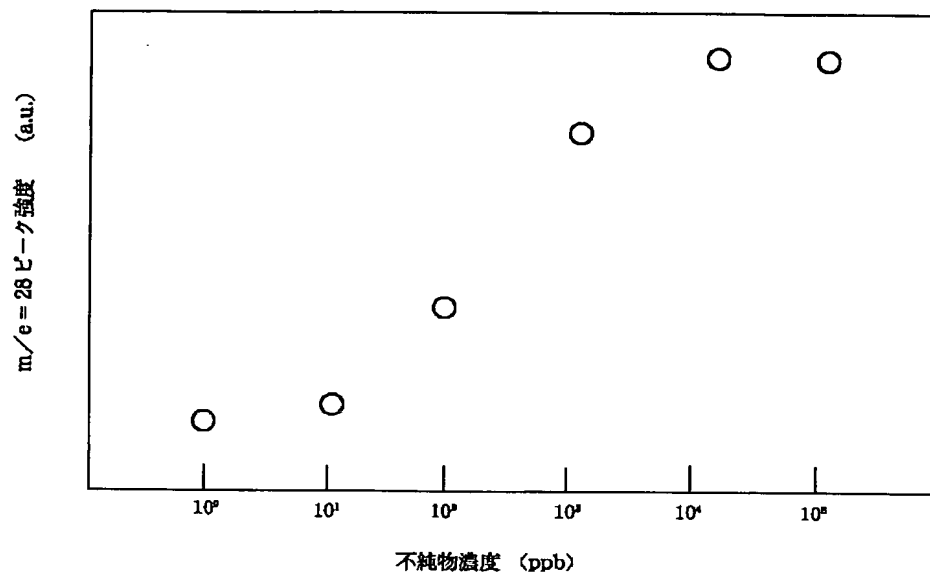
[Drawing 5]



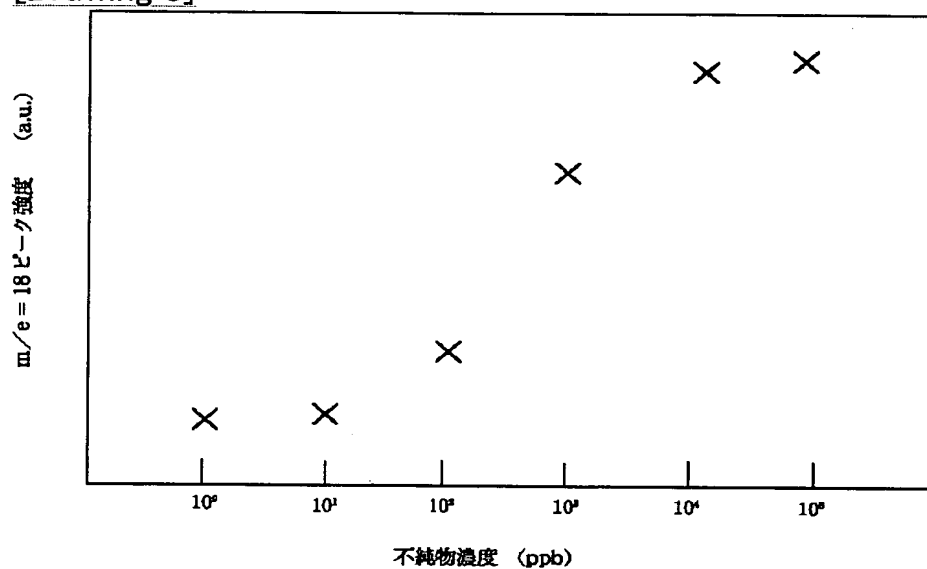
[Drawing 6]



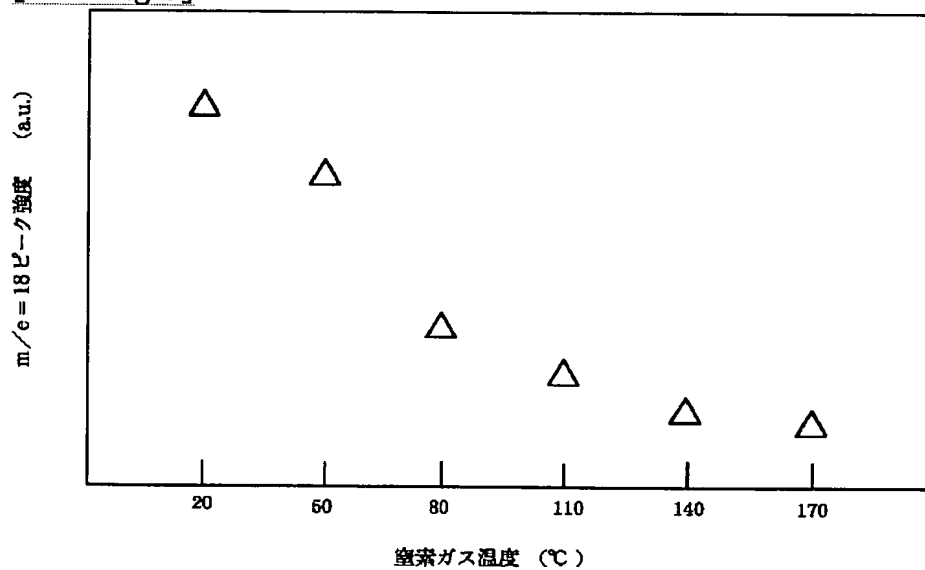
[Drawing 7]



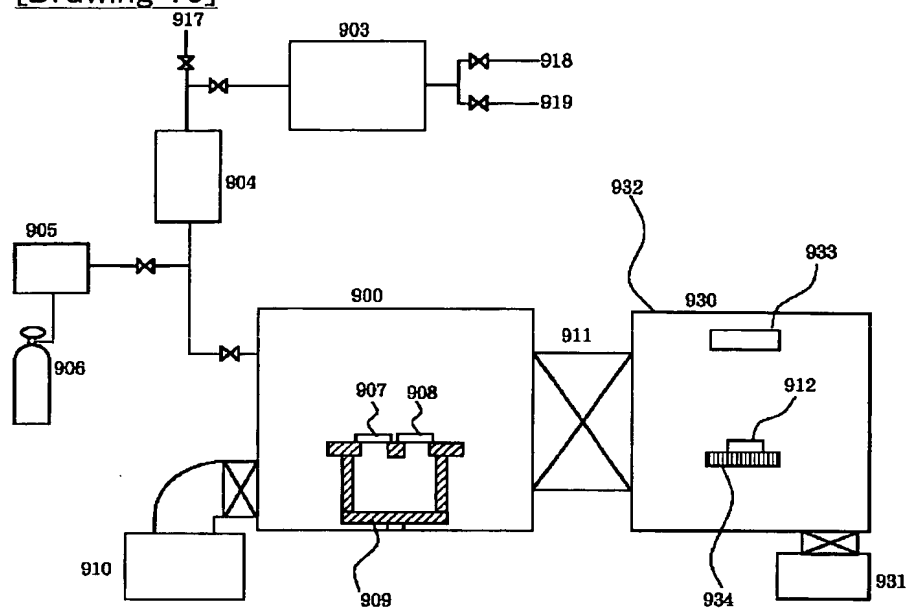
[Drawing 8]



[Drawing 9]



[Drawing 10]



[Translation done.]

[0004] No matter the process, no matter the treatment being performed on a substrate, it is necessary to maintain the purity of the substrate surface before the treatment. That is to say, it is important to eliminate, as thoroughly as possible, the organic impurities and water molecules which exist on the surface of the substrate. Should the degree of purity be lowered or be unstable, the variations in the properties of the substrate after each process of the treatment will be larger. As a result, there was the problem that the yield of various manufactured products was lowered after undergoing each process.

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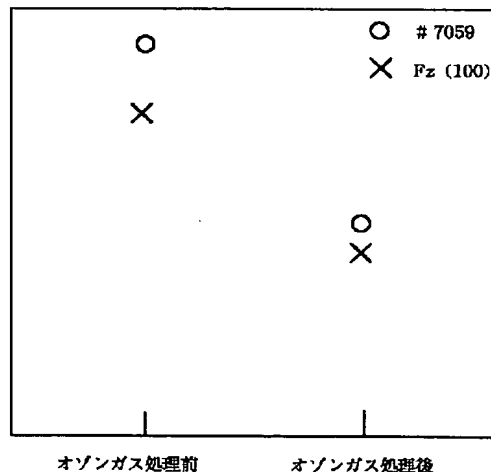
(54) 【発明の名称】 基体の処理方法及び処理装置

(57) 【要約】

【課題】 本発明は、基体から除去した有機物が再付着せず、処理工程を増やすことなく効率的に水分子を除去でき、処理した基体上に薄膜を形成する前に各種乾燥処理の工程が不要で、処理により基体表面の粗さ増大や平坦性の低下が少なく、かつ、基体の材質依存性が少ない基体の処理方法及び処理装置を提供する。

【解決手段】 本発明の基体の処理方法は、少なくとも排気手段を備えた気密室に配設された基体が、オゾンガス又は／及び加熱された気体に曝されることを特徴とする。また、前記オゾンガス又は加熱された気体に含まれる不純物濃度は、10ppb以下が好ましい。さらに、前記加熱された気体の温度は、80℃以上が望ましい。本発明の基体の処理装置は、上述した基体の処理方法を用いたことを特徴とする。また、処理後の基体上に薄膜を形成することを特徴とする。

○/× = 23 レーザ強度 (a.u.)
(CO₂に起因する)



【特許請求の範囲】

【請求項1】 少なくとも排気手段を備えた気密室に配設された基体が、オゾンガスに曝されることを特徴とする基体の処理方法。

【請求項2】 少なくとも排気手段を備えた気密室に配設された基体が、加熱された気体に曝されることを特徴とする基体の処理方法。

【請求項3】 少なくとも排気手段を備えた気密室に配設された基体が、オゾンガス及び加熱された気体に曝されることを特徴とする基体の処理方法。

【請求項4】 前記オゾンガスに含まれる不純物濃度が、10ppb以下であることを特徴とする請求項1又は3に記載の基体の処理方法。

【請求項5】 前記加熱された気体に含まれる不純物濃度が、10ppb以下であることを特徴とする請求項2又は3に記載の基体の処理方法。

【請求項6】 前記加熱された気体の温度が、80℃以上であることを特徴とする請求項2、3又は5のいずれか1項に記載の基体の処理方法。

【請求項7】 前記加熱された気体が、希ガス、窒素ガス、オゾンガス又は水素ガスから選択される1つのガスであることを特徴とする請求項2、3、5又は6のいずれか1項に記載の基体の処理方法。

【請求項8】 請求項1乃至7のいずれか1項に記載の基体の処理方法を用いたことを特徴とする基体の処理装置。

【請求項9】 少なくとも排気手段を備えた気密室及び処理室を有する基体の処理装置において、前記気密室で請求項1乃至7のいずれか1項に記載の基体の処理方法を行った基体上に、前記処理室で薄膜を形成することを特徴とする基体の処理装置。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】 本発明は、基体の処理方法及び処理装置に係る。より詳細には、基体の表面上に付着した有機系不純物又は／及び水分子を除去する基体の処理方法及び処理装置に関する。

【0002】

【従来の技術】 近年、表示装置分野及び半導体分野においては、さらなる大容量・高密度化が望まれている。このとき、これらの分野では、微細表示技術及び微細回路技術などを実現する必要がある。

【0003】 上記技術を達成するため、基体に対して、減圧（真空）雰囲気中では各種の成膜工程、エッチング工程、及びクリーニング工程が、大気雰囲気中では各種の組立工程、洗浄工程、及びエッチング工程が行われている。

【0004】 何れの工程においても、基体に対して何らかの処理を行う場合には、その処理前の基体表面を清浄に保持する必要がある。すなわち、基体表面上に存在す

る有機系不純物や水分子を極力除去することが大切である。この清浄度が低下したり又は不安定な場合には、各工程の処理を終えた基体の諸特性はバラツキが大きくなってしまふ。その結果、各工程を経て製造される各種製品の歩留まりが低下するという問題があった。

【0005】 従来、上記問題に対処する方法としては、次の技術が知られている。

① 大気中において、純水、各種アルコール、又は各種溶剤を用いて基体をウェット洗浄した後、基体上に薄膜を形成する前に各種乾燥処理を行う方法。

② 大気中において、各種エッチング溶液を用いて基体をウェットエッチングした後、基体上に薄膜を形成する前に各種乾燥処理を行う方法。

③ 真空中において、各種プラズマを用いて基体をドライエッチングする方法。

④ 大気中において、低圧水銀灯等の単一ではないスペクトル（184nmの他、200nm以上の波長がある）からなる紫外線の光源により、大気中に含まれる酸素を分解することによりオゾンが発生させ、このオゾンに基体を曝す方法。

⑤ 真空中において、基体上に薄膜を1層以上堆積して、基体上に新たな清浄面を形成する方法。

【0006】 しかし、上記従来技術には、次のような問題がある。

（1）上記①及び②の技術は、基体上に薄膜を形成する前に各種乾燥処理の工程が必須であり、製造コストが高くなる。また、処理後に大気に暴露されるため、基体表面に有機物や水分子が再付着してしまう。

（2）上記②及び③の技術は、エッチングにより基体表面の粗さが増大し、基体の平坦性も低下するため、基体上に作製した薄膜又は構造物の中に乱れが発生する。

（3）上記④の技術は、基体から除去した有機物のポリマライゼーションが発生し、基体に再付着してしまう。また、減圧下においてはオゾンの発生効率が低い。

（4）上記⑤の技術は、真空中における工程が増え製造コストが高くなり、かつ、基体の材質依存性があるため制約が大きい。

【0007】

【発明が解決しようとしている課題】 本発明は、基体から除去した有機物が再付着しない基体の処理方法及び処理装置を提供することを第1の目的とする。

【0008】 また、本発明は、処理工程を増やすことなく効率的に水分子を除去できる基体の処理方法及び処理装置を提供することを第2の目的とする。

【0009】 さらに、本発明は、処理した基体上に薄膜を形成する前に各種乾燥処理の工程が不要で、処理により基体表面の粗さ増大や平坦性の低下が少なく、かつ、基体の材質依存性が少ない基体の処理方法及び処理装置を提供することを第3の目的とする。

【0010】

【課題を解決するための手段】本発明は、少なくとも排気手段を備えた気密室に配設された基体が、オゾンガス又は／及び加熱された気体に曝されることを特徴とする基体の処理方法に要旨が存在する。

【0011】

【発明の実施の形態】以下では、本発明に係る各請求項の作用に関して説明する。

【0012】請求項1に係る発明では、少なくとも排気手段を備えた気密室に配設された基体がオゾンガスに曝されるため、基体上に存在する有機系不純物の残存量を低減できる。

【0013】請求項2に係る発明では、少なくとも排気手段を備えた気密室に配設された基体が加熱された気体に曝されるため、基体上に存在する水分子の残存量を低減できる。

【0014】請求項3に係る発明では、少なくとも排気手段を備えた気密室に配設された基体がオゾンガス及び加熱された気体に曝されるため、基体上に存在する有機系不純物の残存量と水分子の残存量とを同時に低減できる。

【0015】請求項4に係る発明では、前記オゾンガスに含まれる不純物濃度が、10ppb以下であるため、基体上に存在する有機系不純物の残存量をさらに少なくできる。

【0016】請求項5に係る発明では、前記加熱された気体に含まれる不純物濃度が、10ppb以下であるため、基体上に存在する水分子の残存量をさらに少なくできる。

【0017】請求項6に係る発明では、前記加熱された気体の温度が80℃以上であるため、基体上に存在する水分子の残存量を低減する効果が著しく高まる。その結果、基体の処理時間の短縮化も可能となる。

【0018】請求項7に係る発明では、前記加熱された気体が、希ガス、窒素ガス、オゾンガス又は水素ガスから選択される1つのガスであるため、基体上に存在する水分子の除去作用が高い基体の処理方法が得られる。

【0019】請求項8に係る発明では、請求項1乃至7のいずれか1項に記載の基体の処理方法を用いたため、基体上に存在する有機系不純物の残存量又は／及び水分子の残存量を低減できる基体の処理装置が得られる。

【0020】請求項9に係る発明では、少なくとも排気手段を備えた気密室及び処理室を有する基体の処理装置において、前記気密室で請求項1乃至7のいずれか1項に記載の基体の処理方法を行った基体上に、前記処理室で薄膜を形成するため、構造の安定した薄膜が得られる。

【0021】以下、図面を参照して本発明の実施態様例を説明する。

【0022】(少なくとも排気手段を備えた気密室)本発明に係る気密室は、例えば図2、図3又は図10に示

すように、気密室内に導入されたオゾンガスや加熱された気体を排除できる排気手段を有する。排気手段としては、汎用の各種ポンプを用いて構わない。気密室内を減圧下にする場合は、各種真空ポンプが適宜用いられる。特に、導入するガス流量が多い場合は、複数のポンプを多段接続して用いる。例えば、モレキュラードラッグポンプ(ダイキン製、DMS600ACR)+メカニカルブースタポンプ(エドワーズ製、QMB500)+ドライポンプ(エドワーズ製、QDP80)の組み合わせが挙げられる。この組み合わせでは、ガス流量50SLMで連続使用が可能となる。

【0023】気密室の内壁を構成する材料としては、例えばSUS(316L、304L等)、Al、Ni合金(ハステロイ等)が挙げられる。特に、気密室にオゾンガスを導入する場合は、気密室の内壁は耐オゾンガス性の高い材料で構成する必要がある。このような材料としては、例えばFe₂O₃/SUS-316L(表面にFe₂O₃膜を数十nm形成したSUS-316L)が好適である。O₃により材質が改質せず、かつ、Alに比べてO₃が失活しない点から有利である。

【0024】また、O₃以外の加熱された気体を導入する場合、気密室の内壁を構成する材料としては、例えば(Cr₂O₃、Fe₂O₃)/SUS-316L(電解研磨あるいは複合電解研磨したSUS-316Lの表面にCr₂O₃膜やFe₂O₃膜を設けたもの)、表面粗さRaが0.1μm程度に研磨されたAlが好適に用いられる。表面を構成する材料自体からの不純物ガス放出が少ない点が優れている。

【0025】ガス導入系が気密室に接続される位置は、排気手段が気密室に接続される位置とほぼ対向して設け、かつ、気密室内に導入されたガスが乱流となるようにした方が好ましい。この配置を採用した場合、ガスが滞留することなく、本発明の効果が損なわれないという点から優れている。例えば、図2又は図3に示すような配置が挙げられる。

【0026】上述した気密室にゲートバルブを介してTDS(Thermal Desorption Spectroscopy、昇温脱離ガス分析法)分析室を設けた。TDS分析室では、基板に熱を加えることで、基体表面に付着した有機系不純物や水分子を離脱させ、その離脱ガスを後述する分析装置で定量的に評価した。

【0027】TDS分析室は排気手段を備えており、その排気手段は、基体表面に付着した有機系不純物や水分子を評価分析する雰囲気圧力により、気密室と同様に適宜選択される。

【0028】TDS分析室内の圧力が大気圧近傍で測定する場合には、日立東京エレクトロニクス製のTDS装置(UG-21)を分析室に取り付けた(図2)。基体表面から脱離した有機系不純物や水分子の評価は、TDS装置に付設したAPIMS(Atmospheric Pressure I

onization Mass Spectrometer、大気圧イオン化質量分析装置)を用い、実施した。また、分析中はキャリアガスとしてAr、N₂等を適宜導入した。

【0029】一方、減圧下で測定する場合には、電子科学製のTDS装置(EMD-WA1000K)を分析室に取り付けた(図3)。基体表面から脱離した有機系不純物や水分子の評価は、TDS装置に付設したQMS(Quadrupole Mass Analyzer、四重極質量分析器)を用い、実施した。

【0030】(基体)基体としては、例えば、ガラス基板(ホウケイ酸無アルカリガラス、ソーダガラス、石英等)、Siウェハ基板(Fz(100)、Cz(100)等)、セラミックス基板(Al₂O₃、SiC等)が挙げられる。また、これらの基体上に各種皮膜を設けたものも含む。各種皮膜としては、例えば、Al、Mo、Ta、SiN、TiN、a-Si等が挙げられる。さらに、これらの膜を積層して形状加工することで前記基体上に、電子素子又は回路、磁気素子又は回路、及び光学素子又は回路などを配設したものも本発明に係る基体の一例である。

【0031】電子素子又は回路を有する基体としては、例えばTFT(Thin Film Transistor)、MOSFET(Metal Oxide Semiconductor Field Emission Transistor)、又は、Diode Capacitorを設けたものが挙げられる。中でも、TFT(Thin Film Transistor)回路を有するLCDセル(Liquid Crystal Display Cell)すなわちアクティブマトリクス型LCDや、透明電極で形成されたセグメント電極及びコモン電極を有するLCDセル(Liquid Crystal Display Cell)すなわち単純マトリクス型LCDを作製するとき、本発明に係る基体の処理方法及び処理装置が好適に用いられる。

【0032】磁気素子又は回路を有する基体としては、例えば磁気ディスク、磁気ヘッド、磁気カードが挙げられる。光学素子又は回路を有する基体としては、例えばLEDや半導体レーザに代表される各種の受発光デバイス、光(光磁気)ディスクが挙げられる。

【0033】(オゾンガス)本発明に係るオゾンガスは、O₃、O₂及びN₂から構成された混合ガスであり、O₃濃度が1ppm~1000ppm(O₃:(O₂+N₂)=1:10⁶~10³)の範囲にあるものが好適に用いられる。O₃、O₂及びN₂以外のガス成分がオゾンガスに含まれる不純物であり、例えばH₂O、CO₂、CH₄が挙げられる。このオゾンガスに含まれる不純物濃度は、1000ppb以下としたとき、TDS測定時の有機系ガス不純物の検出量が大きく減少し、有機系ガス不純物の除去効果が顕著になるため好ましい。また、この不純物濃度を10ppb以下としたとき、有機系ガス不純物の検出量が少ない状態で飽和することから、基体表面に存在する有機系ガス不純物量がほぼ最小値になったと判断できるため、より好ましい。

【0034】本発明におけるオゾンガスは、気密室近傍に配置した市販のO₃生成器(住友精密工業製、SG-O1A1)を一部改良したものに、不純物濃度を1ppb以下のO₂及びN₂を供給して製造した。得られたオゾンガスの不純物濃度は1ppb以下であり、耐オゾンガス仕様の配管を介して気密室に導入した。また、O₃生成器と気密室を結ぶ配管部に、既知の不純物を10ppm添加したN₂ガス供給系とマスフローコントローラを多段に組み合わせたガス希釈系からなる不純物添加系より、適当量の不純物を含有不純物濃度が1ppb以下のオゾンガスに添加して、気密室に導入するオゾンガスに含まれる不純物濃度を100ppm~1ppbの範囲で変化させた。

【0035】(加熱された気体)本発明に係る加熱された気体としては、気体を介して基体に熱を伝えやすく、かつ、基体表面から水分子を除去できるガス種が好ましい。この理由から、希ガス(He、Ar等)、窒素ガス、オゾンガス又は水素ガスから選択される1つのガスが好適に用いられる。

【0036】各加熱された気体以外のガス成分が各加熱された気体に含まれる不純物であり、例えばH₂O、CO₂、CH₄が挙げられる。加熱された気体が窒素ガスの場合、窒素ガスに含まれる不純物濃度は、含有不純物濃度が1ppb以下の窒素ガスの供給系と気密室を結ぶ配管部に、既知の不純物を100ppm添加した窒素ガスの別の供給系とマスフローコントローラを多段に組み合わせたガス希釈系からなる不純物添加系より、適当量の不純物を含有不純物濃度が1ppb以下の窒素ガスに添加して、気密室に導入する窒素ガスに含まれる不純物濃度を100ppm~1ppbの範囲で変化させた。この各加熱された気体に含まれる不純物濃度は、1000ppb以下としたとき、TDS測定時のH₂Oの検出量が大きく減少し、H₂Oの除去効果が顕著になるため好ましい。また、この不純物濃度を10ppb以下としたとき、H₂Oの検出量が少ない状態で飽和することから、基体表面に存在するH₂O量がほぼ最小値になったと判断できるため、より好ましい。

【0037】気体に熱を加える方法としては、例えば気密室へのガス導入配管にヒーターを取り付け、配管を介して間接的に加熱する方法が挙げられる。各加熱された気体の温度は、50℃以上としたとき、TDS測定時のH₂Oの検出量が大きく減少し、H₂Oの除去効果が顕著になるため好ましい。また、また、この温度を80℃以上としたとき、H₂Oの検出量が少ない状態で飽和することから、基体表面に存在するH₂O量がほぼ最小値になったと判断できるため、より好ましい。

【0038】(少なくとも排気手段を備えた気密室及び処理室を有する基体の処理装置)本発明に係る基体の処理装置は、上述した気密室にゲートバルブを介して処理室が設けてある。処理室は少なくとも排気手段を備えて

いる。その排気手段は、気密室内の動作圧力により気密室と同様に適宜選択される。場合によっては、気密室内の動作圧力に依存せず、独立して適宜選択した排気手段を設けても構わない。したがって、処理室内の圧には特に制限は無い。

【0039】処理室内では、気密室において所定の処理を終えた基体上に薄膜が形成されるが、薄膜形成方法は何れであっても構わない。例えば、大気圧下の場合は塗布法、めっき法等、減圧下の場合は蒸着法、スパッタ法、CVD法等が挙げられる。

【0040】形成される薄膜の材料に制限は無いが、薄膜の諸特性や密着性などを制御する目的から、薄膜が形成される際に基体を適宜加熱又は冷却する場合がある。また、薄膜が形成される空間内に電場又は／及び磁場を導入しても構わない。

【0041】(基体の評価方法) 以下では、図2及び図3を用いて本発明に係る基体の評価方法を説明する。

【0042】(1) TDS分析室内の圧力が大気圧近傍の場合

- ① 基体I及びIIを気密室の回転可能な基体ホルダーにセットした。
- ② 常にAr以外の不純物ガス濃度が1ppb以下の高純度Arでパージされて清浄な雰囲気にあるTDS装置に、基体Iを移動させた。
- ③ Arを2l/min供給しながら、基体Iを室温から500℃まで加熱し、その過程で脱離したガスが含まれるキャリアArガスをAPIMSで分析した。
- ④ 基体Iを気密室に移動後、基体I及びIIにオゾンガス又は／及び加熱された気体による処理を施した。
- ⑤ 上記②及び③と同様に、基体IIをTDS装置に移動させ、APIMSで分析した。

【0043】(2) TDS分析室内の圧力が減圧下の場合

- ①' 基体I及びIIを気密室の回転可能な基体ホルダーにセットした。
- ②' 気密室を 1×10^{-6} Torrに減圧後、予め 2×10^{-9} Torrに減圧してあるTDS装置に、基体Iを移動させた。
- ③' 基体Iを室温から500℃まで加熱し、その過程で脱離したガスをQMSで分析した。
- ④' 基体Iを気密室に移動後、基体I及びIIにオゾンガス又は／及び加熱された気体による処理を施した。
- ⑤' 上記②' 及び③' と同様に、基体IIをTDS装置に移動させ、APIMSで分析した。

【0044】(薄膜の評価方法) 本発明に係るAI薄膜の評価項目としては、比抵抗、ヒロック、温純水酸化及び結晶性が挙げられる。これらの各評価方法を以下に記載する。

【0045】[比抵抗] 比抵抗($\Omega \cdot \text{cm}$)は、シート抵抗(Ω/\square)と膜厚(nm)の積から算出した。シー

ト抵抗は、成膜直後の薄膜(基体上)に対して、ナブソン製のHA-6100/RG-1000Eを用いて測定した。その後、薄膜をパターンニング処理して、シート抵抗測定箇所の膜厚をULVAC製のDEKTA-K-3030で測定した。

【0046】[ヒロック] ヒロックは、以下の手順により測定した。

- ① 成膜直後の薄膜(基体上)を光学顕微鏡で観察し、写真を撮った。
- ② 上記①の薄膜(基体ごと)をN₂雰囲気下、400℃で2時間アニール処理した。
- ③ 上記②の処理を終えた薄膜(基体上)を光学顕微鏡で観察し、写真を撮った。
- ④ 上記①と③の写真(倍率:825)に写っているヒロックの数を各々計測し、その差分を求め、単位面積当たりのヒロック増加の有無を調べた。ヒロックサイズが数百nm程度以上のものを計測した。

【0047】[温純水酸化] 温純水酸化は、以下の手順により測定した。

- ① 上記比抵抗で膜厚測定後の薄膜(基体上)を、80℃の温超純水に20分間浸漬した。
- ② 上記①の処理を終えた薄膜(基体上)の膜厚を測定した。浸漬後の膜厚が浸漬前より増加した場合、AIが酸化されてAI₂O₃に変化し体積が増えたと判断した。この酸化状態の有無は、XPS(Xray Photoelectron Spectroscopy、X線光電子分光法)を用いて別途確認した。

【0048】[結晶性] 結晶性は、成膜直後の薄膜が付着した基体を3cm角にカッティング後、理研製のX線回折測定装置で回折強度を測定した。

【0049】

【実施例】以下に実施例をあげて本発明をより詳細に説明するが、本発明がこれら実施例に限定されることはない。

【0050】(実施例1) 本例では、図2に示した気密室内に基体を配設し、大気圧近傍においてオゾンガスに曝される前後の基体上に存在する有機系不純物の残存量を、気密室にゲートバルブを介して設けたTDS分析室において調べた。TDS分析室では、基板に熱を加えることで、基体表面に付着した有機系不純物を離脱させ、その離脱ガスをAPIMSを用いて定量的に評価した。基体としては、Siウェハ(Fz(100))又はガラス(#7059)を用いた。

【0051】以下では、実験手順にしたがって説明する。

(1) 次の①及び②に示した処理を順次行い、2枚の基体を洗浄した。

- ① 基体が入ったカセット(材質:テフゼル)を超純水(比抵抗:18M Ω)浸漬させ、超純水中にメガソニック(0.8MHz)を10分間加えた。

② 上記①の処理を終えた基体をスピン乾燥（850 rpm、2分間）した。

（2）上記（1）の処理を終えた2枚の基体が入ったカセットを気密室内へ挿入した後、1枚の基体は未処理のままTDS分析室へ移し、基体表面に付着した有機系不純物を離脱させ、その離脱量をAPIMSで測定した。

（3）他の1枚の基体は、気密室内に次の条件でオゾンガスを導入することでオゾンガスに曝した。

・供給ガス：O₃100ppm/O₂:N₂=1:4

・オゾンガスの不純物濃度：1ppb以下

・気密室内の内圧：760Torr

・供給時間：10min

（4）上記（3）の処理を終えた基体をTDS分析室へ移し、上記（2）と同様に基体表面に付着した有機系不純物を離脱させ、その離脱量を測定した。

【0052】図1は、オゾンガスに曝す前後の基体表面から離脱した有機系不純物量（質量数28）の測定結果である。図1から、オゾンガスに曝した基体の表面では、基体の材質に依存せず、有機系不純物が除去されていることが分かった。

【0053】（実施例2）本例では、図3に示した気密室内に基体を配設し、減圧下にある気密室において基体をオゾンガスに曝した点が実施例1と異なる。TDS分析室も気密室と同レベルの減圧下とし、基板に熱を加えることで、基体表面に付着した有機系不純物を離脱させ、その離脱ガスをQMSを用いて定量的に評価した。他の点は、実施例1と同様とした。

【0054】以下では、実験手順にしたがって説明する。

（1）実施例1の工程（1）と同様に2枚の基体を洗浄した。

（2）上記（1）の処理を終えた2枚の基体が入ったカセットを気密室内へ挿入した後、気密室内を 1×10^{-6} Torrまで減圧した。

（3）1枚の基体は未処理のまま、前もって 2×10^{-9} Torrまで減圧してあるTDS分析室へ移し、基体表面に付着した有機系不純物を離脱させ、その離脱量をQMSで測定した。

（4）他の1枚の基体は、気密室内に次の条件でオゾンガスを導入することでオゾンガスに曝した。

・供給ガス：O₃100ppm/O₂:N₂=1:4

・オゾンガスの不純物濃度：1ppb以下

・気密室内の内圧：700Torr

・供給時間：10min

（5）上記（4）の処理を終えた基体を前もって 2×10^{-9} Torrまで減圧してあるTDS分析室へ移し、上記（2）と同様に基体表面に付着した有機系不純物を離脱させ、その離脱量を測定した。

【0055】図4は、オゾンガスに曝す前後の基体表面から離脱した有機系不純物量（質量数28）の測定結果

である。図4から、減圧下においてもオゾンガスに曝した基体の表面では、基体の材質に依存せず、有機系不純物が除去されていることが分かった。

【0056】（実施例3）本例では、図2に示した気密室内に基体を配設し、大気圧近傍において加熱された気体に曝される前後の基体上に存在する水分子の残存量を、気密室にゲートバルブを介して設けたTDS分析室において調べた。加熱された気体としては、50℃の窒素ガスを用いた。

【0057】TDS分析室では、基板に熱を加えることで、基体表面に付着した水分子を離脱させ、その離脱ガスをAPIMSを用いて定量的に評価した。基体としては、Siウェハ（Fz（100））又はガラス（#7059）を用いた。他の点は、実施例1と同様とした。

【0058】以下では、実験手順にしたがって説明する。

（1）実施例1の工程（1）と同様に2枚の基体を洗浄した。

（2）上記（1）の処理を終えた2枚の基体が入ったカセットを気密室内へ挿入した後、1枚の基体は未処理のままTDS分析室へ移し、基体表面に付着した水分子を離脱させ、その離脱量をAPIMSで測定した。

（3）他の1枚の基体は、気密室内に次の条件で窒素ガスを導入することで窒素ガスに曝した。

・供給ガス：N₂（温度50℃、流量50SLM）

・窒素ガスの不純物濃度：1ppb以下

・気密室内の内圧：760Torr

・供給時間：10min

（4）上記（3）の処理を終えた基体をTDS分析室へ移し、上記（2）と同様に基体表面に付着した水分子を離脱させ、その離脱量を測定した。

【0059】図5は、加熱された窒素ガスに曝す前後の基体表面から離脱した水分子量（質量数18）の測定結果である。図5から、加熱された窒素ガスに曝した基体の表面では、基体の材質に依存せず、水分子が除去されていることが分かった。

【0060】（実施例4）本例では、図3に示した気密室内に基体を配設し、減圧下にある気密室において基体を加熱された窒素ガスに曝した点が実施例3と異なる。TDS分析室も気密室と同レベルの減圧下とし、基板に熱を加えることで、基体表面に付着した水分子を離脱させ、その離脱ガスをQMSを用いて定量的に評価した。他の点は、実施例3と同様とした。

【0061】以下では、実験手順にしたがって説明する。

（1）実施例1の工程（1）と同様に2枚の基体を洗浄した。

（2）上記（1）の処理を終えた2枚の基体が入ったカセットを気密室内へ挿入した後、気密室内を 1×10^{-4} Torrまで減圧した。

(3) 1枚の基体は未処理のまま、前もって 2×10^{-9} Torrまで減圧してあるTDS分析室へ移し、基体表面に付着した水分子を離脱させ、その離脱量をQMSで測定した。

(4) 他の1枚の基体は、気密室内に次の条件で窒素ガスを導入することで窒素ガスに曝した。

- ・供給ガス：N₂（温度50℃、流量50SLM）
- ・窒素ガスの不純物濃度：1ppb以下
- ・気密室の内圧：700Torr
- ・供給時間：10min

(5) 上記(4)の処理を終えた基体を前もって 2×10^{-9} Torrまで減圧してあるTDS分析室へ移し、上記(2)と同様に基体表面に付着した水分子を離脱させ、その離脱量を測定した。

【0062】図6は、加熱された窒素ガスに曝す前後の基体表面から離脱した水分子量（質量数18）の測定結果である。図6から、減圧下においても加熱された窒素ガスに曝した基体の表面では、基体の材質に依存せず、水分子が除去されていることが分かった。

【0063】実施例3及び4では、加熱された気体として窒素ガスを用いたが、窒素ガスの代わりに希ガス（例えばHe、Ar、Xe等）、オゾンガス又は水素ガスを用いても同様の作用があることが別途確認された。特に、加熱された気体としてオゾンガスを採用した場合は、実施例1又は2と実施例3又は4の効果を同時に実現できる。すなわち、基体の表面上に存在する有機系不純物と水分子を同時に除去できるため、処理工程の短縮化を図ることができ、低コスト化の可能な基体の処理方法が得られた。

【0064】（実施例5）本例では、オゾンガスに含まれる不純物濃度を1ppm～1ppbの範囲で変えた点が実施例1と異なる。基体としては、ガラス（#7059）を用いた。他の点は、実施例1と同様とした。

【0065】図7は、オゾンガスに含まれる不純物濃度と、処理後の基体表面に残存する有機系不純物の量を測定した結果である。図7から、1ppm以下としたとき、TDS測定時に基体表面から離脱する有機系不純物の量が急激に減少することから、基体表面に残存する有機系不純物の量が減少することが分かった。また、10ppb以下としたとき、基体表面から離脱する有機系不純物の量の減少する傾きが大幅に緩和することから、基体表面に残存する有機系不純物の量がほぼ最小値に近づいたと判断した。

【0066】（実施例6）本例では、加熱された窒素ガスに含まれる不純物濃度を1ppm～1ppbの範囲で変えた点が実施例3と異なる。基体としては、ガラス（#7059）を用いた。他の点は、実施例3と同様とした。

【0067】図8は、加熱された窒素ガスに含まれる不純物濃度と、処理後の基体表面に残存する水分子の量を

測定した結果である。図8から、1ppm以下としたとき、TDS測定時に基体表面から離脱する水分子の量が急激に減少することから、基体表面に残存する水分子の量が減少することが分かった。また、10ppb以下としたとき、基体表面から離脱する水分子の量の減少する傾きが大幅に緩和することから、基体表面に残存する水分子の量がほぼ最小値に近づいたと判断した。

【0068】（実施例7）本例では、窒素ガスの加熱温度を室温（約20℃）～400℃の範囲で変えた点が実施例3と異なる。基体としては、ガラス（#7059）を用いた。他の点は、実施例3と同様とした。

【0069】図9は、窒素ガスの温度と、処理後の基体表面に残存する水分子の量を測定した結果である。図9から、50℃以上としたとき、基体表面から離脱する水分子の量が急激に減少することから、基体表面に残存する水分子の量が減少することが分かった。また、80℃以上としたとき、基体表面から離脱する水分子の量の減少する傾きが大幅に緩和することから、基体表面に残存する水分子の量がほぼ最小値に近づいたと判断した。

【0070】（実施例8）本例では、図10に示した気密室において加熱された気体に表面を曝された基体上に、処理室で薄膜を形成し、作製した薄膜の諸特性を調べた。

【0071】加熱された気体としては窒素ガスを、基体としてはガラス（#7059）を、薄膜としてはアルミを用いた。評価した膜特性は、比抵抗、ヒロック、温純水酸化および結晶性である。

【0072】以下では、実験手順にしたがって説明する。

(1) 実施例1の工程(1)と同様の基体洗浄を行った。

(2) 上記(1)の処理を終えた基体が入ったカセットを気密室へ挿入した。

(3) 気密室内を 1×10^{-4} Torrまで真空排気した。

(4) 成膜前処理として、加熱された窒素ガス（温度150℃、流量50SLM）を導入し、排気コンダクタンスを調節することにより、気密室内の圧力を5Torrとし、1分間パージ処理した。

(5) 気密室への窒素ガス供給を停止した後、気密室内を 1×10^{-2} Torrまで真空排気した。

(6) 上記(4)～(5)の工程を4回繰り返した。

(7) 気密室内を 5×10^{-7} Torrまで真空排気した。

(8) 別途 5×10^{-7} Torrまで真空排気してある処理室へ、気密室から基体を移動した。

(9) 処理室において、電力密度1.5W/cm²の周波数13.56MHzの高周波電力（又は、電力密度4W/cm²の直流電力）をカソードに供給し、プラズマを発生させ、加熱された基体（150℃）上に厚さ20

0 nmのアルミ膜を形成した。

【0073】上記(1)～(9)の工程により作製した試料は、試実8と呼称した。

【0074】(比較例1)本例では、実施例8における基体の前処理、すなわち実施例8の工程(3)～(6)を削除した。他の点は、実施例8と同様とした。

【0075】本例で作製した試料は、試比1と呼称した。

【0076】表1は、上述した実施例6と比較例1で作製した各試料に対して行った、比抵抗、ヒロック、温純水酸化、及び結晶性に関する評価結果である。

【0077】

【表1】

評価項目	試実6	試比6
比抵抗	2.8 $\mu\Omega$	3.0 $\mu\Omega$
ヒロック	確認されず	確認された
温純水酸化	酸化されず	酸化を確認
結晶性	50 cps/nm	2 cps/nm

【0078】表1から、試比1に比べて試実6の方が安定した薄膜であることが分かった。したがって、表示装置分野及び半導体分野などにおいて、各種薄膜を作製する場合、本発明に係る基体の処理方法を用いることで膜構造の安定性を高められると判断した。

【0079】また一般的に、結晶性が改善されたことから、本発明に係る基体の処理方法は、処理面における粗さ増大や平坦性の低下がほとんど無いと判断した。

【0080】本例では、構造の簡単な単層膜を用いて本発明に係る基体の処理方法を検討したが、多層構造や複雑な構造を有する各種の半導体デバイスやTFT-LCDなどを作製する工程においても有効であることは言うまでもない。

【0081】

【発明の効果】以上説明したように、本発明によれば、基体から除去した有機物が再付着せず、処理工程を増やすことなく効率的に水分子を除去でき、処理した基体上に薄膜を形成する前に各種乾燥処理の工程が不要で、処理により基体表面の粗さ増大や平坦性の低下が少なく、かつ、基体の材質依存性が少ない基体の処理方法及び処理装置が得られる。

【図面の簡単な説明】

【図1】本発明の実施例1に係る大気圧近傍においてオゾンガスに曝す前後の基体表面から離脱した有機系不純物の量の測定結果を示すグラフである。

【図2】本発明の実施例1に係る気密室及びTDS分析

室からなる基体の処理装置の模式的断面図である。

【図3】本発明の実施例2に係る気密室及びTDS分析室からなる基体の処理装置の模式的断面図である。

【図4】本発明の実施例2に係る減圧下においてオゾンガスに曝す前後の基体表面から離脱した有機系不純物の量の測定結果を示すグラフである。

【図5】本発明の実施例3に係る大気圧近傍において加熱された窒素ガスに曝す前後の基体表面から離脱した水分子の量の測定結果を示すグラフである。

【図6】本発明の実施例4に係る減圧下において加熱された窒素ガスに曝す前後の基体表面から離脱した水分子の量の測定結果を示すグラフである。

【図7】本発明の実施例5に係るオゾンガスに含まれる不純物濃度と、処理後の基体表面に残存する有機系不純物の量の測定結果を示すグラフである。

【図8】本発明の実施例6に係る加熱された窒素ガスに含まれる不純物濃度と、処理後の基体表面に残存する水分子の量の測定結果を示すグラフである。

【図9】本発明の実施例7に係る窒素ガスの温度と、処理後の基体表面に残存する水分子の量の測定結果を示すグラフである。

【図10】本発明の実施例8に係る気密室及び処理室からなる基体の処理装置の模式的断面図である。

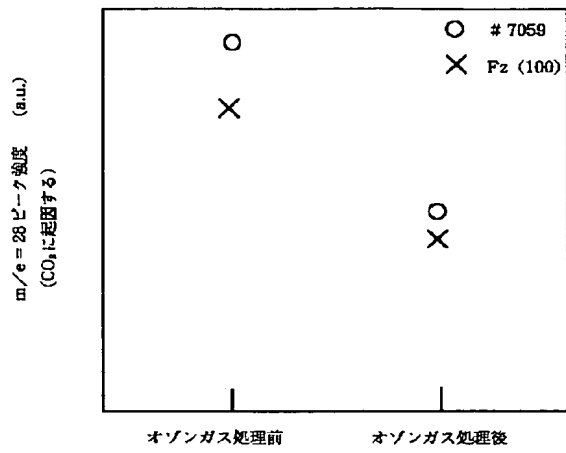
【符号の説明】

200、300、900 気密室、
 201、301、901 TDS装置、
 202 APIMS、
 203、303、903 O₃生成器、
 204、304、904 温ガス発生機構、
 205、305、905 ガス希釈器、
 206、306、906 標準ガスボンベ、
 207、307、907 基体I、
 208、308、908 基体II、
 209、309、909 基体ホルダー(カセット)、
 210、310、320、910、931 ターボ分子ポンプ、
 211、311、911 ゲートバルブ、
 212、312 TDS測定時の基体、
 213、313 TDS装置の基体ホルダー、
 214 赤外線ランプ、
 215 高純度Ar導入ライン、
 216 TDS装置とAPIMS間のArライン、
 217、218、317、318、917、918 高純度N₂ライン、
 219、319、919 高純度O₂ライン、
 302 QMS、
 314 赤外線導入装置、
 912 処理室内の基体、
 930 処理室、
 932 Arライン、

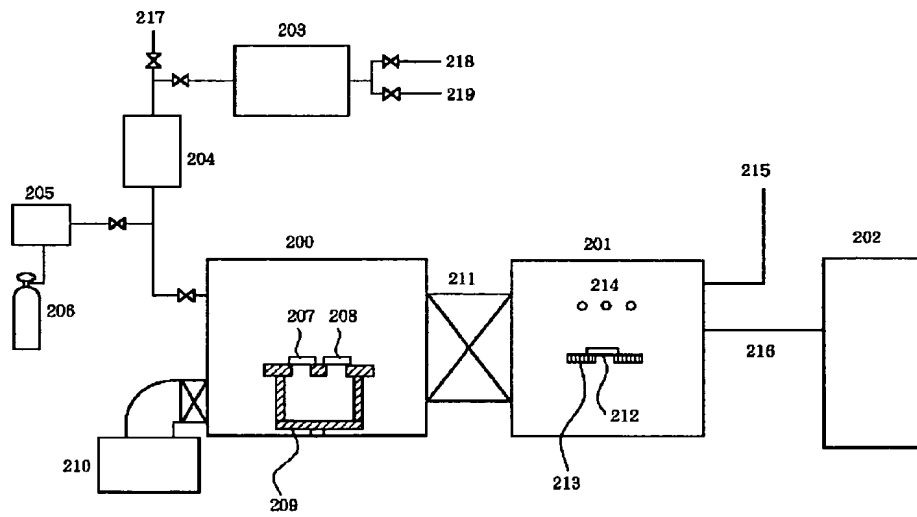
933 A1ターゲット、

934 基板ホルダー兼カソード。

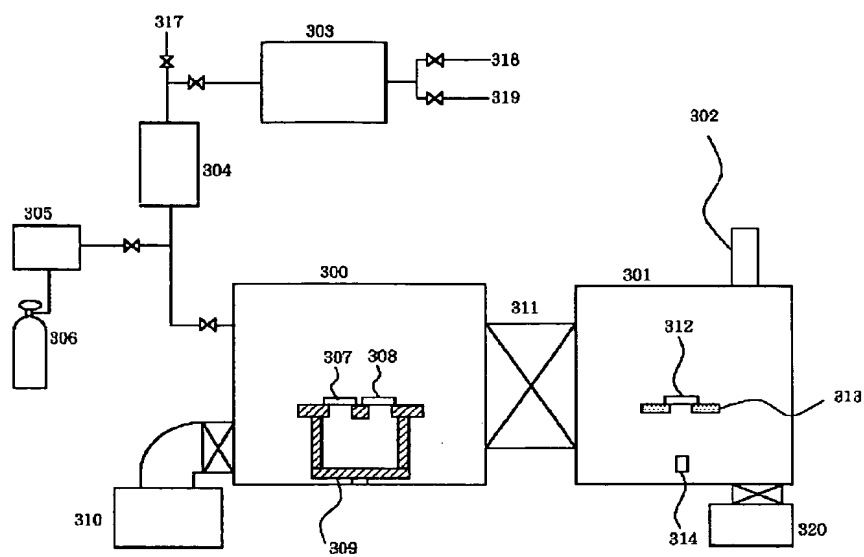
【図1】



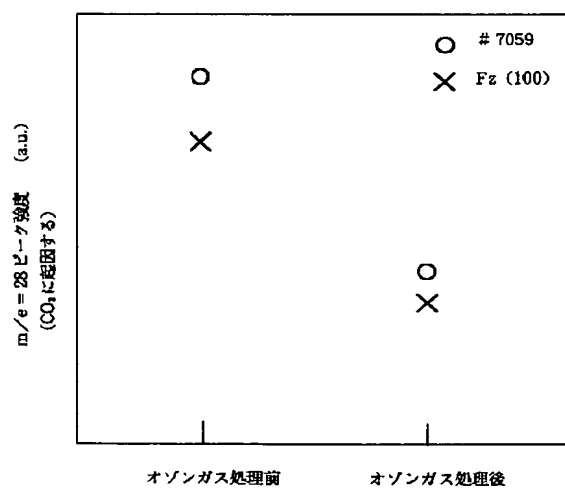
【図2】



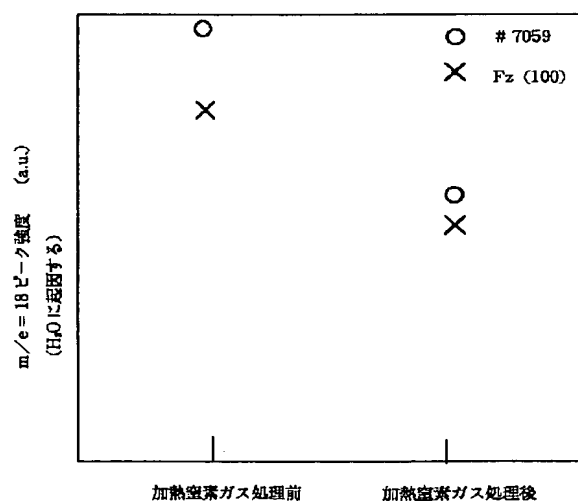
【図3】



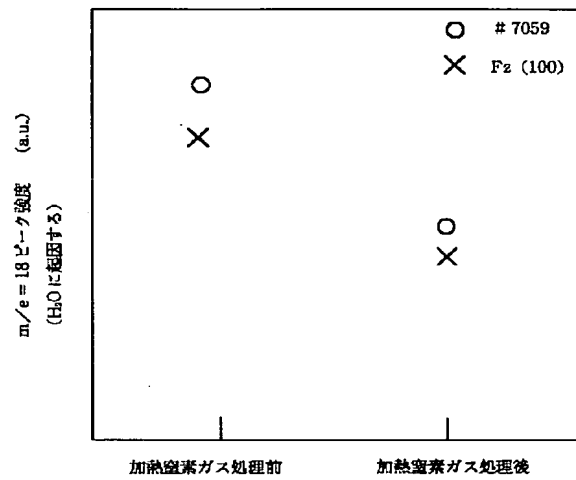
【図4】



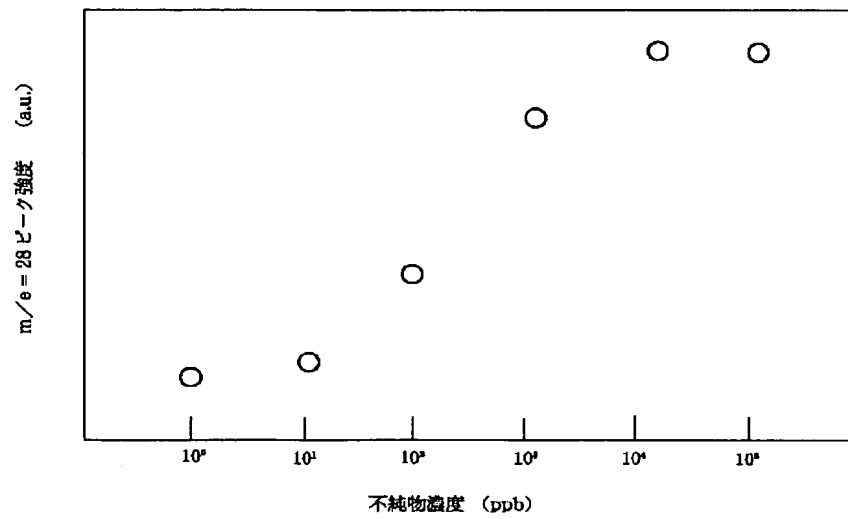
【図5】



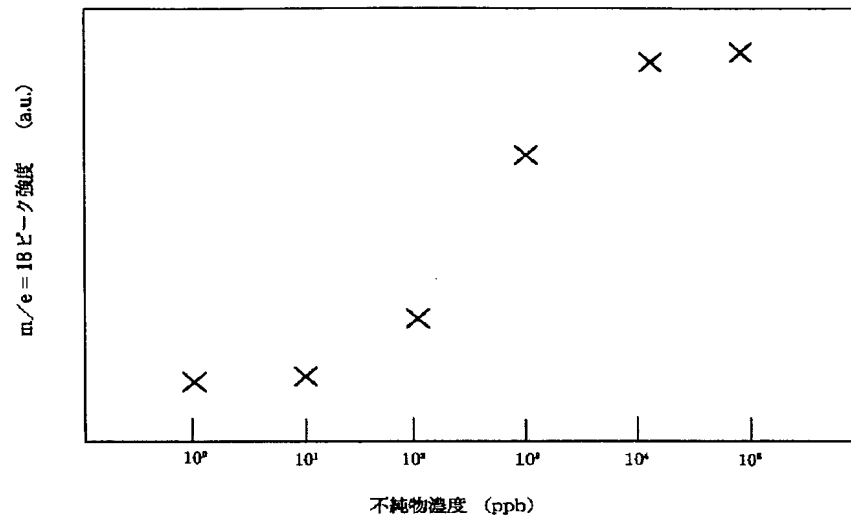
【図6】



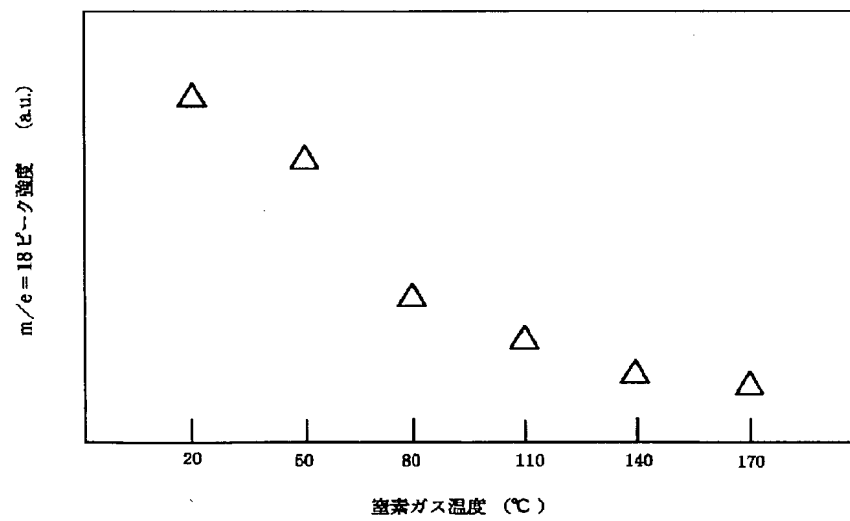
【図7】



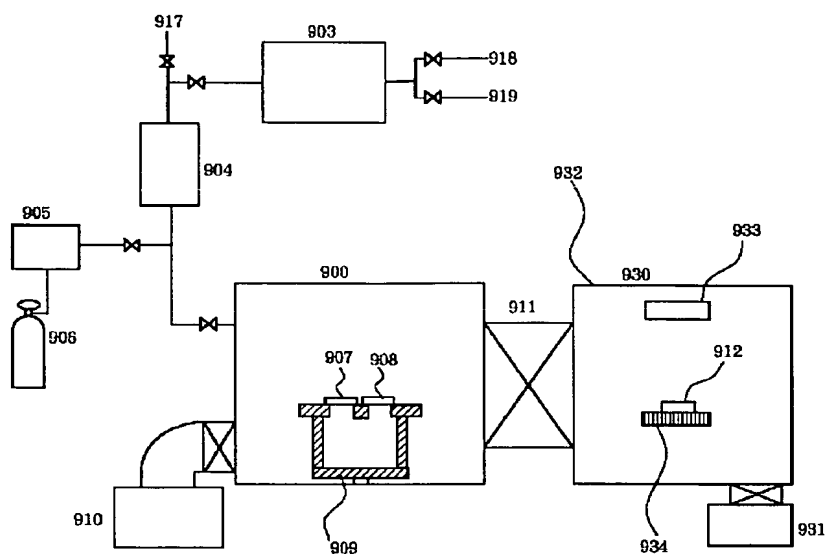
【図8】



【図9】



【図10】



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